



GB00/3275

**BEST AVAILABLE COPY**

The Patent Office  
Concept House  
Cardiff Road  
Newport

South Wales  
NP10 8QQ  
REC'D 11 OCT 2000

WIPO PCT

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.

Signed

Dated 18 September 2000

**PRIORITY  
DOCUMENT**

SUBMITTED OR TRANSMITTED IN  
COMPLIANCE WITH RULE 17.1(a) OR (b)

# Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1.	Your reference	BP-08-1365		
2.	Patent application number (The Patent Office will fill in this part)	9921504.8		
3.	Full name, address and postcode of the or of each applicant (underline all surnames)	THE MORGAN CRUCIBLE COMPANY PLC Morgan House Madeira Walk Windsor Berkshire SL4 1EP		
	Patents ADP number (if you know it)			
	If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM 06832232001		
4.	Title of the invention	HIGH TEMPERATURE RESISTANT SALINE SOLUBLE FIBRES		
5.	Name of your agent (if you have one)	PHILLIPS & LEIGH		
	"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)	7 Staple Inn Holborn London WC1V 7QF		
	Patents ADP number (if you know it)	0001289001 ✓		
6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number	Country	Priority application number (if you know it)	Date of filing (day / month / year)
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing (day / month / year)	
8.	Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d))	Yes		

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

Continuation sheets of this form -

Description 7

Claim(s) 2

Abstract -

Drawing(s) 3 + 3

10. If you are also filing any of the following, state how many against each item.

Priority documents -

Translation of priority documents -

Statement of inventorship and right to grant of a patent (Patents Form 7/77) -

Request for preliminary examination and search (Patents Form 9/77) -

Request for substantive examination (Patents Form 10/77) -

Any other documents (please specify) -

11. I/We request the grant of a patent on the basis of this application.

Signature

*J. C. Boff*

Date

10<sup>th</sup> September 1999

12. Name and daytime telephone number of person to contact in the United Kingdom J. C. Boff 0171 405 0133

### Warning

*After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.*

### Notes

- If you need help to fill in this form or you have any questions, please contact the Patent Office on 0645 500505.
- Write your answers in capital letters using black ink or you may type them.
- If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.
- If you have answered 'Yes' Patents Form 7/77 will need to be filed.
- Once you have filled in the form you must remember to sign and date it.
- For details of the fee and ways to pay please contact the Patent Office.

## HIGH TEMPERATURE RESISTANT SALINE SOLUBLE FIBRES

This invention relates to high temperature resistant saline soluble fibres and particularly relates to calcium-magnesium-silicate fibres.

Calcium-magnesium-silicate fibres are known for example from WO89/12032, WO93/15028 and WO94/15883.

WO89/12032 first disclosed a broad class of fire-resistant saline soluble fibres.

WO93/15028 showed that a class of the fibres of WO89/12032 were usable at temperatures up to 1000°C or more.

WO94/15883 showed that some of the fibres of WO93/15028 had higher use temperatures still, of up to 1260°C or more, and indicated that such fibres needed a SiO<sub>2</sub> excess (defined as the amount of SiO<sub>2</sub> remaining after crystallisation of CaO, MgO and any ZrO<sub>2</sub> as silicates) of greater than 21.8mol%.

WO97/16386 while falling in the general class of calcium-magnesium-silicate fibres looked to low calcium fibres to show use temperatures of 1260°C or more.

It is apparent that there are regions of the CaO-MgO-SiO<sub>2</sub> and CaO-MgO-SiO<sub>2</sub>-ZrO<sub>2</sub> composition fields within which high temperature performance fibres can be made and other regions where they cannot.

The applicants have now found a new and narrow range of compositions that are usable at temperatures of 1200°C or more and even 1250°C or 1260°C or more, and yet fall outside the scope of WO94/15883 and WO97/16386. These compositions preferably have little or no zirconia.

Accordingly the present invention provides a fibre having a maximum use temperature of 1200°C or more in which the amount of MgO in mol% is greater than the amount of CaO in mol % and which comprises:-

$\text{SiO}_2 > 64.25 \text{ wt\%}$

$\text{CaO} > 18 \text{ wt\%}$

$\text{MgO} < 17 \text{ wt\%}$ .

However, no claim is made to fibres having a  $\text{SiO}_2$  excess as specified of greater than 21.8mol%.

Further features of the invention are apparent from the appended claims.

The excess  $\text{SiO}_2$  figure is calculated by treating all of the CaO as being bound as  $\text{CaO.MgO.2SiO}_2$ ; all of the  $\text{ZrO}_2$  as being bound as  $\text{ZrO}_2.\text{SiO}_2$ ; and the remaining MgO as being bound as  $\text{MgO.SiO}_2$ . The applicants also assume that any  $\text{Al}_2\text{O}_3$  crystallises as  $\text{Al}_2\text{O}_3.\text{SiO}_2$ . Any remaining  $\text{SiO}_2$  is called the excess  $\text{SiO}_2$ .

The invention is illustrated by way of example in the following description with reference to the drawings in which

Fig 1 is a graph showing linear shrinkage with temperature for blankets comprising the fibres A4-2 and A4-3 of Table 1 below.

Fig. 2 is a graph showing shrinkage through the height of blankets comprising the fibres A4-2 and A4-3 of Table 1 below.

Fig. 3 is a graph showing shrinkage of preforms produced from fibres A4-1, A4-2 and A4-3 of Table 1 below.

Table 1 shows compositions extracted from WO89/12032, WO93/15028, WO94/15883, and WO97/16386 together with A4, a target composition fibre having the composition:-

SiO<sub>2</sub> 65 wt%

CaO 19.5 wt%

MgO 15.5 wt%

and A4-1, A4-2, and A4-3, which are analysed fibre samples.

The fibres extracted from the data of WO89/12032 (referred to as Manville fibres), WO93/15028, WO94/15883, and WO97/16386 (referred to as Unifrax fibres) are those for which the SiO<sub>2</sub> excess as specified is less than 21.8mol% and for which the amount of MgO in mol% is greater than the amount of CaO in mol%.

A4-1 was produced as bulk fibre; A4-2 was produced as needled blanket having a density of approximately 96 kg.m<sup>-3</sup>; and A4-3 was produced as needled blanket having a density of approximately 128 kg.m<sup>-3</sup>.

In Table 1 shrinkages are indicated from the documents concerned or, for A4-1, A4-2, and A4-3, from measuring the shrinkage of vacuum formed preforms of the fibres concerned.

Table 1															
Fibre	Shrinkage at 1260°C	Composition wt%						Composition mol%						SiO <sub>2</sub> Excess	
		CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>	Others	CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>		SiO <sub>2</sub>
924		19.78	14.54	0.66	2.57		61.32		19.99	20.44	0.30	1.43		57.84	15.68
SW-A1		20.50	15.20		1.10		63.70		20.16	20.79	0.00	0.59		58.46	16.92
B5	6.00	19.90	15.10	0.10	0.20		64.20	0.40	19.71	20.80	0.05	0.11		59.34	18.68
757		20.92	15.22	0.00	0.20		62.60		20.79	21.04	0.00	0.11		58.06	16.13
A4-1	1.86	20.20	15.50		0.26		64.60	0.07	19.76	21.10	0.00	0.14		59.00	18.00
A4-2	3.25	20.20	15.50		0.30		64.50	0.06	19.78	21.11	0.00	0.16		58.95	17.89
A4-3	2.76	20.20	15.60		0.28		64.70	0.07	19.72	21.18	0.00	0.15		58.95	17.9
A4 target		19.50	15.50				65.00		19.17	21.20	0.00	0.00		59.64	19.27
SW-A2		21.40	15.40		0.80		60.80		21.40	21.42	0.00	0.44		56.74	13.49
SW-A		20.50	15.50		3.30		59.30		20.66	21.73	0.00	1.83		55.78	11.56
932		21.60	15.65	0.11	1.50		59.85		21.58	21.75	0.05	0.82		55.80	11.60
Manville104		17.70	16.30		1.83		64.10		17.49	22.40	0.00	0.99	0.00	59.11	18.23
B9	5.00	18.10	17.10	0.10	0.20		64.40	0.20	17.72	23.29	0.04	0.11		58.84	17.69
971		23.92	17.36	0.05	0.74		56.82		23.56	23.79	0.02	0.40		52.23	4.47
B8	6.90	18.80	17.90	0.30	0.20		63.00	0.20	18.30	24.24	0.13	0.11		57.23	14.46
B16	4.30	15.10	18.10	0.30	0.10	0.10	66.00	0.20	14.78	24.65	0.13	0.05	0.07	60.31	20.69
A2-12		16.55	18.00	0.05	0.33		63.56		16.37	24.76	0.02	0.18		58.67	17.34
A2-30		16.06	18.21	0.00	0.40		63.68		15.89	25.07	0.00	0.22		58.82	17.64

Table 1																	
Fibre	Shrinkage at 1260°C	Composition wt%							Composition mol%							SiO <sub>2</sub> Excess	
		CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>	Others	CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>			
A2-23		18.59	18.78	0.05	0.48		60.20		18.37	25.82	0.02	0.26		55.53			11.06
71		24.04	19.66	0.00	0.20		54.68		23.44	26.67	0.00	0.11		49.77			-0.45
A2-19		18.48	19.74	0.00	0.54		58.71		18.29	27.18	0.00	0.29		54.24			8.47
A2-21		13.74	19.98	0.13	0.34		64.16		13.51	27.34	0.06	0.18		58.90			17.81
Unifrax comp 13	13.10	0.77	16.90	34.10	0.97		46.40		0.92	28.11	18.55	0.64		51.78			3.56
A2-24		13.62	22.74	0.08	0.31		61.38		13.25	30.79	0.04	0.17		55.76			11.51
Manville105		9.74	23.10		2.15		65.10		9.38	30.95	0.00	1.14	0.00	58.53			17.05
Manville79		8.67	24.00		0.02		67.20		8.27	31.86	0.00	0.01	0.00	59.86			19.71
A2-25		10.99	24.18	0.07	0.33		62.36		10.66	32.64	0.03	0.18		56.48			12.97
Unifrax comp 14	23.40	0.89	21.70	24.10	0.90		51.90		0.98	33.18	12.05	0.54		53.24			6.49
A2-35		8.88	24.88	0.47	0.29		64.12		8.56	33.37	0.21	0.15		57.71			15.41
Manville78		6.43	26.50				67.10		6.07	34.80	0.00	0.00	0.00	59.13			18.25
A2-34		6.63	26.20	0.80	0.23		64.85		6.37	35.01	0.35	0.12		58.15			16.29
Unifrax comp 15	25.00	0.77	25.50	16.90	0.76		55.70		0.80	36.82	7.98	0.43		53.96			7.93
Manville77		4.02	28.70		0.59		66.10		3.79	37.68	0.00	0.31	0.00	58.22			16.44
Unifrax 42	2.00	0.30	29.75	0.58	0.62		68.63		0.28	38.92	0.25	0.32		60.23			20.47



Table 1																
Fibre	Shrinkage at 1260°C	Composition wt%							Composition mol%							SiO <sub>2</sub> Excess
		CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>	Others	CaO	MgO	ZrO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	SiO <sub>2</sub>		
Manville106		2.70	29.70		1.56		65.60		2.54	38.94	0.00	0.81	0.00	57.71	15.41	
Manville80		1.60	30.10				68.40		1.49	39.02	0.00	0.00	0.00	59.49	18.98	
Manville71		3.12	30.10		1.15		65.40		2.92	39.26	0.00	0.59	0.00	57.23	14.45	
Manville76		3.12	30.10		1.15		65.40		2.92	39.26	0.00	0.59	0.00	57.23	14.45	
Unifrax 47	7.70	0.25	30.26	0.01	1.86		67.53		0.23	39.56	0.00	0.96		59.24	18.48	
Unifrax 40	9.20	0.27	30.57	0.58	0.92		67.52		0.25	39.90	0.25	0.47		59.13	18.25	
765		3.90	35.07	0.00	2.12		57.78		3.62	45.26	0.00	1.08		50.04	0.07	

It can be seen that the fibres according to the present invention show lower shrinkage at 1260°C than do the extracted fibres other than fibre Unifrax 42 which has a radically different composition.

In Figs. 1-3, graphs show the shrinkage characteristics of the fibres A4-1, A4-2, and A4-3 after 24 hours exposure to the indicated temperatures. it can be seen that the fibres are readily usable at temperatures of 1200°C or more.

Table 2 below shows the results of solubility tests on the fibres in physiological saline solution indicating that the fibres are soluble in body fluids. (See WO94/15883 for a discussion of methods of measuring solubility). Pairs of results are indicated for separate tests on each sample as is a mean total solubility.

Table 2					
Fibre type	Solubility (ppm)				
	CaO	MgO	SiO <sub>2</sub>	Total	Mean Total
A4-1	102	115	171	388	383
	105	110	162	377	
A4-2	105	116	172	393	395
	114	117	166	397	
A4-3	114	123	166	403	411
	114	128	177	419	

A typical range of compositions for fibres of the present invention would be

SiO<sub>2</sub> 65 ± 0.5 wt%

CaO 20 ± 0.5 wt%

MgO 15 ± 0.5 wt%.

## CLAIMS

DISCLAIMER: - No claim is made to fibres having a  $\text{SiO}_2$  excess as specified of greater than 21.8mol%.

1. A fibre having a maximum use temperature of 1200°C or more which comprises:-  
 $\text{SiO}_2 > 64.25 \text{ wt\%}$   
 $\text{CaO} > 18 \text{ wt\%}$   
 $\text{MgO} < 17 \text{ wt\%}$   
and in which the amount of MgO in mol% is greater than the amount of CaO in mol %.
2. A fibre as claimed in claim 1 and comprising:-  
 $\text{CaO} < 21 \text{ wt\%}$ .
3. A fibre as claimed in claim 2 and comprising:-  
 $\text{CaO} < 20.5 \text{ wt\%}$ .
4. A fibre as claimed in any of claims 1 to 3 and comprising:-  
 $\text{CaO} > 19 \text{ wt\%}$ .
5. A fibre as claimed in claim 4 and comprising:-  
 $\text{CaO} > 19.5 \text{ wt\%}$
6. A fibre as claimed in any of claims 1 to 4 and comprising:-  
 $\text{MgO} > 14.25 \text{ wt \%}$ .
7. A fibre as claimed in claim 6 and comprising:-  
 $\text{MgO} > 14.75 \text{ wt \%}$ .

8. A fibre as claimed in claim 7 and comprising:-  
MgO > 15.25 wt %.
9. A fibre as claimed in any preceding claim and comprising:-  
MgO < 16 wt % .
10. A fibre as claimed in any preceding claim and comprising:-  
SiO<sub>2</sub> 65 ± 0.5 wt%  
CaO 20 ± 0.5 wt%  
MgO 15 ± 0.5 wt%.
11. A fibre as claimed in claim 10 and comprising:-  
SiO<sub>2</sub> 64.5 - 64.7 wt%  
CaO 19.5 - 20.2 wt%  
MgO 15.5 - 15.6 wt%.





